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X-Ray Spectrochemical and X-Ray Diffraction Analyses of Copper-Platinum Alloys

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FOREWORD

This report was prepared by the Physics Laboratory. The work was initiated under Project 7360, "Materials Analysis and Evaluation Techniques," and Task 736005, "Compositional, Atomic, and Molecular Analysis." It was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, with W.L. Baun acting as project engineer.

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ABSTRACT

Results are shown for X-ray spectrochemical analysis and X-ray diffraction analysis of alloys in the copper-platinum alloy system. Lattice parameters are shown for disordered alloys from copper through platinum. Results from X-ray spectrochemical analyses of the alloys are shown and several methods of analysis are discussed. The results show that both zinc and gold are acceptable internal standards for the Cu-Pt alloys and that a coherent scattering internal standard can be used where only a quick approximate answer is required.

This report has been reviewed and is approved.

A handwritten signature in cursive script, reading "Freeman F. Bentley".

FREEMAN F. BENTLEY
Chief, Analytical Branch
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INTRODUCTION

During an investigation of ordering in the alloy system copper-platinum (Cu-Pt), two techniques were used for the quantitative analysis of these alloys. One method determined alloy composition by X-ray emission spectroscopy; the other method approximated the composition by accurate measurement of lattice parameters from well-annealed alloy samples. Once a complete set of alloys were run and calibration curves were drawn, either of these two methods was much faster than the conventional wet chemical technique.

This report summarizes the results of these X-ray studies on the Cu-Pt alloy system. Data will be shown only for disordered alloys. Results on long-range order and high-temperature diffractometer studies will be presented in a separate report.

X-RAY DIFFRACTION

The phase diagram for the Cu-Pt alloy system is shown in figure 1. Measurements of electrical conductivity (or resistivity) (refs 6,7,9), temperature coefficient of resistance (ref 7), and lattice parameter measurements (refs 6,7,10) covering the entire range of composition prove that Cu and Pt form a continuous series of solid solutions at high temperatures. As the phase diagram indicates, order-disorder transformations take place in a wide range of compositions. A further study of this ordering is in progress along with a study at the order-disorder boundary using a high temperature diffractometer mount.

Samples and Equipment

All alloy samples used in this particular work were obtained from C. Hartley of the (Directorate of Materials & Processes). They had been carefully melted and homogenized, and were in the form of $\frac{1}{4}$ inch rods. Sections of the rod were removed, pressed into pellets about one inch in diameter, and subsequently annealed above 1000°C in argon to remove stress caused by pressing. These specimens were then used for diffractometer measurements. In addition, powdered samples were obtained by filing each original rod, passing a magnet through the filings, and annealing the filings above 1000°C to relieve stress due to filing. The final powder diffraction specimen was prepared by placing 325-mesh or smaller particles in thinwalled 0.7 mm glass capillaries.

Patterns for the disordered alloys were recorded using a Philips Electronics, Inc., diffractometer and scintillation counter with pulse-height analyzer. Nickel-filtered Cu radiation was used in all cases. Powder specimens were used in a 2-radian Siemens powder camera using Cu radiation.

Lattice Parameter Measurement

Figure 2 shows a plot of lattice parameters determined by the authors along with those shown by Schneider and Esch (ref 10). Agreement is relatively good. Lattice parameters plotted here (also shown in table 1) were obtained from diffractometer values on samples scanned at $\frac{1}{4}$ degree per minute. Lattice parameters were determined by using

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$K\alpha_1$, $K\alpha_2$, and $K\beta$ values for the (400), (331), and (420) lines of the f.c.c. alloys. These values were then extrapolated to $\cos^2 \theta = 0$ and $\frac{1}{2} \left[\frac{\cos^2 \theta}{\sin \theta} + \frac{\cos^2 \theta}{\theta} \right] = 0$.

These extrapolations gave resulting lattice parameters which agreed within about .0002 Å. The values shown in table 1 are those obtained from the extrapolation to $\frac{1}{2} \left[\frac{\cos^2 \theta}{\sin \theta} + \frac{\cos^2 \theta}{\theta} \right] = 0$.

The resulting lattice parameter curve shown in figure 2 was used to determine composition on unknowns in the 40 to 60 atomic percent Pt region. Values varied from the theoretical by only $\pm .5$ percent. No standards had to be run with each sample after the original curve was plotted (another advantage of this method over the conventional).

The appendix shows the X-ray powder diffraction data for Cu, Pt, and some of the disordered alloys of the Cu-Pt system. These values are from film patterns taken in a 2-radian camera. The Cu and Pt shown are the starting materials for the preparation of alloys.

X-RAY SPECTROCHEMICAL ANALYSIS

Samples and Equipment

For the X-ray spectrochemical analysis a Siemens Crystalloflex IV X-ray Spectrograph was operated at 45 KV and 22 MA using a tungsten target X-ray tube, LiF analyzing crystal, and a scintillation counter with a detector voltage of 885 volts. The "a" beam collimator was used for maximum sample illumination with 6-mm exit and receiving slits and a 0.15-degree Soller slit. The samples were contained in a 5-ml polyethylene liquid sample holder with Mylar window. A fixed count procedure was used throughout with 10^4 counts taken each of five times on all emission peaks and 10^3 counts each of five times on the background.

All standard solutions were prepared from pure metals, reagent grade acids, and de-ionized water.

All samples were made with an elemental concentration of approximately 6.0 mg/ml. The standard solutions were prepared so as to produce samples differing in elemental composition by 10 percent. These solutions were run using (1) no internal standard, (2) a $W_{L\beta}$ internal standard, (3) Zn internal standard, and (4) Au internal standard. Calibration charts were prepared for each of these standard solutions by plotting element/standard ratio versus mg/ml of the element and element/standard ratio versus percent of total sample.

The experimental samples were dissolved in a 1:1 solution of HCL and HNO_3 at a concentration of approximately 6.0 mg/ml, and run with the standards listed above. The results of these analyses will be discussed in more detail later.

Discussion and Results

Background

X-ray emission spectroscopy has provided a rapid analytical technique of relatively

high precision. Several deviations from proportionality exist, however, and the usefulness of any spectroscopic method depends upon how conveniently and satisfactorily these deviations can be handled. The object of this investigation is to determine a method best suited to minimizing these deviations in the samples under investigation. Liebhafsky, Pfeiffer, Winslow, and Zemany (ref 8) group these deviations into three general classes. Class I includes absorption and enhancement effects. A constant absorption effect does not affect analytical results but, rather, is the change of absorption effects from one sample to another that leads to deviations. Absorption effects can be calculated, and even a simplified treatment is ordinarily adequate for most analyses. Enhancement effects are more complex and difficult to handle. Enhancement arises when an emission wavelength of one element of the sample excites the emission wavelength of another. Class II effects are the result of heterogeneity of the sample. Solid samples must be thoroughly mixed or homogeneous in nature. The use of liquid samples reduces this problem. Class III deals primarily with instability and fluctuations of equipment. Of these three classes, the most prevalent to the analyst are the effects of Class I. Class II deviations are solved largely by the establishment of sound sample preparation techniques, and Class III by good experimental techniques.

Absorption and enhancement (Class I) provide the more difficult problems, the solution of which depends upon the method selected for analysis. To discuss this problem in more detail, several accepted techniques shall be considered.

Emission Intensity Versus Composition

The intensity versus composition relationship of an element in a matrix is not a linear function but, rather, a curve, the attitude of which depends upon the relative absorption or enhancement coefficients. Curve "a" in figure 1 shows the plot of a heavy metal in a light matrix and shows the intensity rising rapidly and falling off sharply as the composition becomes rich in the heavy component. In curve "b" the absorption coefficient of the matrix is slightly less than that of the element, and in curve "c" the absorption coefficient of the matrix is slightly greater. This is a simple graphic example of absorption and enhancement effects.

To compensate for such effects, the internal standard techniques have been used, of which there are several accepted methods.

1. The addition of a known amount of another element.

An element is chosen whose emission line occurs in the same region of the spectrum, thereby showing the same or closely similar absorption and enhancement. It is important that in the element chosen there are no other higher order emission lines in the same area of the spectrum.

2. The addition of a known amount of the same element.

In some cases it is difficult to provide another element that will furnish the requirement as outlined above. In such a case, a weighed portion of the same element can be added and by measuring the intensity of the element, I_E , and of the element plus the additional percentage, $I_E + \%$, the composition may be calculated by:

$$I_E/I_E + \% = C/(C + \%)$$

3. Coherent scattering of primary radiation.

In the coherent scattering method, the intensity of an emission peak of the characteristic tube target radiation is measured near an emission peak of the element to be analyzed.

4. Incoherent scattering.

Properly chosen wavelengths of incoherent scattering, primarily in the low wavelength portion of the spectrum, may satisfactorily serve as an internal standard. Because of the increased amount of scatter, this method may be particularly applicable to the analysis of liquid samples.

All of these methods may compensate for absorption and enhancement effects as well as for sample and instrumental variations.

Technique

The Cu-Pt binary alloys were submitted in the form of $\frac{1}{4}$ inch rods. The samples were prepared by filing, and the filings were passed over a magnet to remove any possible iron contamination. A portion of each sample was then weighed (approximately 0.16 gm). To ensure that all samples were in the same oxidation state, all samples were dissolved in a 1:1 solution of HCL and HNO₃.

The analysis of solutions has several advantages over the analysis of solids. Principal among these is the homogeneity of the sample, subsequent dilution of matrix effects, ease of addition and dispersion on an added internal standard, and the ability to dilute or concentrate to any convenient concentration. There are disadvantages, however, that must be considered which may interfere with intensity measurements, including density effects, high background levels, and incoherent scattering.

Methods of Analysis

Four different methods of analysis were used to determine the best method for determination of the samples.

1. Intensity versus composition.

Coherent scattered radiation varies with the atomic number to the 1st or 2nd power, dependent upon the magnitude of the atomic number. Elements of low atomic numbers, because of the separation of the electrons, scatter X-rays as if the electrons were isolated and acting independently. As the atomic number increases, the electrons become closer spaced and scatter the electrons nearly in phase, resulting in an increased total intensity. This effect increases as the concentration of the element increases.

The intensities of the CuK α , and PtL α , were measured and plotted against percent composition using standard solutions. The plot was similar to Birks (ref 2) in figure 1, but showed little usefulness for accurate precise quantitative analysis and, as an analytical method, was abandoned.

2. Coherent primary radiation.

Using only coherently scattered characteristic tube radiation, with the atomic number to the 1st power, the ratio varies. This is obviously an improvement over straight intensity or background ratio measurement.

Scattered radiation is composed of two parts, coherent and incoherent, as expressed in the Andermann-Kemp (ref 1) equation:

$$I_o = S_a/2\mu_o + S_c/\mu_c + \mu_o$$

where;

I_o = observed scattered radiation intensity

μ_o = abs. coeff. of the observed wavelength

μ_c = abs. coeff. of the Compton wavelength

S_a = coherent scattered radiation intensity

S_c = Compton scattered radiation intensity

Since the characteristic X-ray tube target radiation is measured at the coherently scattered radiation peak, the second part of the equation becomes insignificant, and the equation simplifies to:

$$I_o = S_a/2\mu_o$$

Compton and Allison (ref 4) give the equation for the coherent scattered radiation intensity as:

$$S_a = I_e Z F^2 \text{ (low Z elements)}$$

where:

I_e = electron scatter intensity as a constant

Z = atomic number

F^2 = atomic structure factor

The absorption coefficient, μ , is also dependent on the atomic numbers of the elements:

$$\mu = C \cdot N/A \cdot Z^4 \lambda^3$$

where:

C = constant

N = Avagadros number

A = atomic weight

Z = atomic number

λ = wavelength observed

The usual fluorescent intensity equation of an atom is:

$$I_f = t/\mu_i + \mu_o$$

where:

I_f = intensity of fluorescent radiations

t = emission coeff. (constant for given element)

μ_i = abs. coeff. for incident radiation

μ_o = abs. coeff. for observed radiation

Relating intensities to atomic numbers:

$$I_o \sim Z^{-3}$$

and

$$I_f \sim Z^{-4}$$

thus:

$$I_f/I_o \sim Z^{-1}$$

Therefore, intensity ratio measurements should be less sensitive to matrix effects than either straight intensity measurements or background ratio measurements.

However, intensity measurements must be free from interference of other spectral radiations emitted by the sample, and is the most severe limitation of the technique. A recent paper and thorough discussion of the subject concerning coherent scattering as an internal standard is that by T. J. Cullen (ref 5).

Tungsten radiation provides a number of strong L lines, providing a choice of line measurement. For the Cu-Pt analysis, tungsten L_{β} , was found to be most convenient as a standard. A ratio of WL_{β} , and Cu and Pt was calculated and plotted against percent composition and against concentration in mg/ml. The results of the analysis can be found in table 2. These and other results will be discussed in more detail later in this report.

3. Addition of an internal standard.

The introduction of an internal standard into an analytical determination compensates for the effects of absorption and enhancement, instrumental variables, and sample variations upon the intensity measurements. The most important factor is that it compensates for absorption and enhancement effects. The simplicity of X-ray spectra makes it possible to preselect a satisfactory internal standard. Birks (ref 2) states that the internal standard is selected on the basis of absorption and enhancement coefficients and would therefore be an element $Z \pm 1$ to the element being analyzed where Z is the atomic number. Blank and Heller (ref 3) state that the selection should be the element with the closest excitation potential. The proper selection, as illustrated by the two standards selected in this study, is not quite so simple.

Consider the possible effects of adding a standard to a solution containing two elements, Cu and Pt. In considering the influence of differential absorption, we are concerned with the location of the absorption edge of the standard relative to the two emission lines;

while considering the enhancement or differential excitation, we are concerned with the characteristic lines of the standard relative to the absorption edge positions of the two elements. Consider figures 2 and 3. The absorption edge of the standard being on the high wavelength side of the two spectral lines will partially absorb both with little effect to their relative intensities. An absorption edge falling to the short wavelength side will affect neither (figure 2).

An absorption edge occurring between two spectral lines (figure 3) will partially absorb the line on the short wavelength side, while the other remains unaffected. This will obviously change the ratio of the recorded intensities.

The added element may also enhance the intensity of the spectral line. Consider figures 4 and 5. The spectral line of the standard in figure 4 occurs on the short wavelength side of both absorption peaks. The enhancement of element 1 depends upon the proximity of the standard's spectral line. If $\Delta\lambda$ is not great enough, the intensity of element 1 will be increased and the ratio will be altered. Should the spectral line fall on the long wavelength side, neither will be affected, and the ratio will be unaltered.

In the case illustrated in figure 5, the spectral line falls between the two absorption edges, element 1 is unaffected, while element 2 is partially excited; the intensity is increased, and ratio is altered.

Obviously, the conditions illustrated in figures 3 and 5 are to be avoided, while those of 2 and 4 are acceptable if the choice of standard is carefully made.

In many cases, a separate standard is needed for each element because any possible combination of spectral lines of the elements are too far removed from each other to give satisfactory results with a single standard. A more ideal situation would be one in which a single standard would be acceptable for both. In this study, the Cu and Pt showed a good proximity of spectral lines, and an effort was made to choose a single standard that would give good results for both determinations. Au and Zn were chosen as possible standards. Solution of Au and Zn were made in concentrations equal to that of the standard solutions. To each 10-ml standard solution, 0.5 ml of the internal standard solution was added. The intensity ratios were calculated and calibration curves were made, plotting intensity ratios versus percent composition and versus mg/ml for both the Au and the Zn standards.

Applying the material previously discussed, neither the Zn nor the Au causes any absorption of the emission intensity of either Cu or Pt (figure 6), since the absorption edges of both fall to the short wavelength side of both Cu and Pt. Figure 7 shows the AuL_{α} line occurring between the absorption edges of Cu and Pt, a situation previously indicated as undesirable. However, in this case, it may be acceptable inasmuch as the absorption edges of Cu and Pt are widely separated ($\Delta\lambda = 0.31\text{\AA}$). The zinc lines as shown appear to produce no difficulty. The AuL_{α} line may partially excite the CuK series, but on the other hand, the AuL_{α} line may also excite the PtL series, and the ratio will remain unaltered. On this basis, it was decided that Au would also be tried as a possible standard. Zn seemed, at least schematically, to be a satisfactory standard for both Cu and Pt.

Element Composition Determination

The results of the study are listed in table 2. The results show that both Zn and Au are acceptable as internal standards for the Cu-Pt binary alloys, and that the WL_{β} internal

standard, while giving less precise values, is acceptable for determinations which demand less accuracy.

One interesting outcome of the investigation, as yet not completely answered, is also illustrated in table 2. When the element/standard intensity ratios were applied to the ratio/percentage composition calibration curves, somewhat erratic answers resulted. Since the samples were in solution form, and since the experimental procedures were carefully controlled, this effect could have been the result of some unknown absorption/enhancement effects inherent within the samples themselves. This problem was successfully resolved by applying the element/standard ratios to the ratio mg/ml calibration curves. In this procedure, the elemental composition at Cu and Pt were determined and collectively taken as 100 percent since the sum of the two was less than the original sample weight in all cases. By taking this sum as 100 percent and then calculating the percentage of each constituent, good agreement to actual percentages was found. This again indicates some absorption/enhancement effect of the samples themselves which caused a low mg/ml reading for each while the ratio of one to the other remained correct.

The procedure described in this report is obviously applicable only to a system wherein all the components are known, and would not be applicable to the determination of an element or elements in a system where some percentage of the composition consists of other components. The complexity of such a system would require the identification of all the components and a determination of the effects due to the added matrix materials.

The results of this study also indicate that the selection of an internal standard need not be governed by atomic number or necessarily by excitation potential, but rather by the proximity of its absorption edges and emission lines of any series to the positions of the analytical emission lines and absorption edges of the elements to be determined.

Although established procedures of spectroscopy analysis exist and in many cases are readily applicable, each determination is significantly different from another so that an investigation as to the best method of analysis, and the best choice of internal standard, sample concentrations, and experimental procedure are deemed warranted.

The sample percentages listed as "Act. %" in table 2 are the percentages of the initial constituents of the alloy and the deviations shown are the deviations from their values. Possible nonhomogeneity in the rod would make these deviations unrealistic.

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APPENDIX
X RAY POWDER DATA FOR DISORDERED Cu-Pt ALLOYS

d, Å	Cu (hkl)	I/I ₁	d, Å	10.2 at. % Pt (hkl)	I/I ₁	d, Å	25.2 at. % Pt (hkl)	I/I ₁
2.067	(111)	vs	2.094	(111)	vs	2.121	(111)	vs
1.796	(200)	s	1.816	(200)	s	1.840	(200)	s
1.273	(220)	s	1.287	(220)	s	1.304	(220)	s
1.086	(311)	s	1.098	(311)	s	1.114	(311)	s
1.043	(222)	ms	1.052	(222)	m	1.067	(222)	ms
0.9036	(400)	m	0.9125	(400)	m	0.9247	(400)	m
0.8292	(331)	s	0.8375	(331)	s	0.8491	(331)	ms
0.8085	(420)	s	0.8165	(420)	s	0.8279	(420)	ms
d, Å	40.6 at. % Pt (hkl)	I/I ₁	d, Å	70.9 at. % Pt (hkl)	I/I ₁	d, Å	78.7 at. % Pt (hkl)	I/I ₁
2.151	(111)	vs	2.201	(111)	vs	2.209	(111)	vs
1.870	(200)	s	1.910	(200)	s	1.920	(200)	s
1.323	(220)	s	1.352	(220)	s	1.359	(220)	s
1.130	(311)	s	1.155	(311)	vs	1.160	(311)	vs
1.083	(222)	ms	1.106	(222)	ms	1.112	(222)	ms
0.9391	(400)	m	0.9602	(400)	ms	0.9650	(400)	ms
0.8618	(331)	ms	0.8814	(331)	s	0.8856	(331)	s
0.8402	(420)	ms	0.8592	(420)	s	0.8634	(420)	s
			0.7848	(422)	s	0.7888	(422)	s
d, Å	86.1 at. % Pt (hkl)	I/I ₁	d, Å	90.75 at. % Pt (hkl)	I/I ₁	d, Å	100.0 at. % Pt (hkl)	I/I ₁
2.227	(111)	vs	2.246	(111)	vs	2.266	(111)	vs
1.927	(200)	s	1.947	(200)	s	1.961	(200)	s
1.366	(220)	s	1.376	(220)	s	1.388	(220)	s
1.168	(311)	vs	1.174	(311)	s	1.183	(311)	s
1.116	(222)	m	1.124	(222)	ms	1.133	(222)	m
0.9697	(400)	m	0.9750	(400)	m	0.984	(400)	m
0.8902	(331)	s	0.8946	(331)	s	0.8995	(331)	s
0.8679	(420)	s	0.8718	(420)	s	0.8799	(420)	s
0.7928	(422)	s	0.7959	(422)	s	0.8006	(422)	s

TABLE I
COMPARISON OF LATTICE PARAMETER ($a_0, \text{\AA}$) MEASUREMENTS FOR
DISORDERED Cu-Pt ALLOYS

at. % Pt	$a_0, \text{\AA}$	at. % Pt	$a_0, \text{\AA}$
100	3.9230	100	3.9158
90.75	3.8976	95.5	3.9014
86.07	3.8846	86.6	3.8805
78.68	3.8658	78.3	3.8554
70.95	3.8440	70	3.8346
58	3.8160	58.5	3.8001
40.60	3.7594	40	3.7485
25.22	3.6974	25	3.6973
10.20	3.6506	10	3.6402
0 (Cu)	3.6163	0 (Cu)	3.6075
Our Data		Schneider and Esch (Ref 10)	

TABLE 2
QUANTITATIVE DETERMINATIONS FROM Cu-Pt ALLOYS

Sample No.	Act. %	Zinc Standard			Gold Standard			Tungsten Standard					
		%	%-Ratio	mg/ml -Ratio	%	%-Ratio	mg/ml -Ratio	%	%-Ratio	mg/ml -Ratio			
		Dev			Dev			Dev					
C O P P E R													
2	3.3	4.2	+0.9	3.4	+0.1	5.2	+1.9	5.4	+2.1	5.1	+1.8	4.5	+1.2
3	4.5	6.8	+2.3	6.3	+1.8	6.5	+2.0	6.5	+2.0	7.4	+2.9	7.1	+2.6
4	8.0	10.0	+2.0	9.2	+1.2	10.8	+2.3	10.5	+2.4	10.2	+2.2	10.1	+2.9
5	12.3	14.7	+2.4	13.9	+1.6	14.0	+1.7	13.0	+0.7	15.4	+3.1	14.6	+2.3
6	19.5	21.3	+1.8	19.2	-0.3	18.5	-1.0	18.9	-0.6	19.4	-0.1	20.5	+1.0
8	33.2	37.0	+3.8	33.8	+0.6	34.0	+0.8	32.9	-0.3	26.4	-6.8	34.4	+1.2
9	50.0	52.9	+2.9	48.4	-1.6	57.0	+7.0	50.5	+0.5	57.8	+7.8	51.0	+1.0
10	74.0	79.9	+5.9	73.2	-0.8	81.0	+7.0	74.7	+0.7	81.4	+7.4	79.3	+5.3
P L A T I N U M													
2	96.6			96.6	+0.0	100.0	+3.4	94.6	-2.0	94.2	-2.4	95.5	-1.1
3	95.5			93.7	-1.8	94.5	+1.0	93.4	-2.1	94.9	-0.6	92.9	-2.6
4	92.0			90.8	-1.2	94.6	+2.6	89.5	-2.5	90.4	-1.6	89.9	-2.1
5	87.7			86.2	-1.5	93.5	+5.8	86.9	-0.8	89.6	+1.9	85.4	-2.3
6	80.5			80.8	+0.3	78.5	-2.0	81.1	+0.6	75.7	-4.8	79.5	-1.0
8	66.8			66.2	-0.6	68.4	+1.6	67.0	+0.2	62.6	-4.2	65.6	-1.2
9	50.0			51.6	+1.6	55.9	+5.9	49.5	-0.5	55.1	+5.1	49.0	-1.0
10	26.0			25.8	-0.2	27.5	+1.5	25.2	-0.8	22.8	-3.2	20.7	-5.2

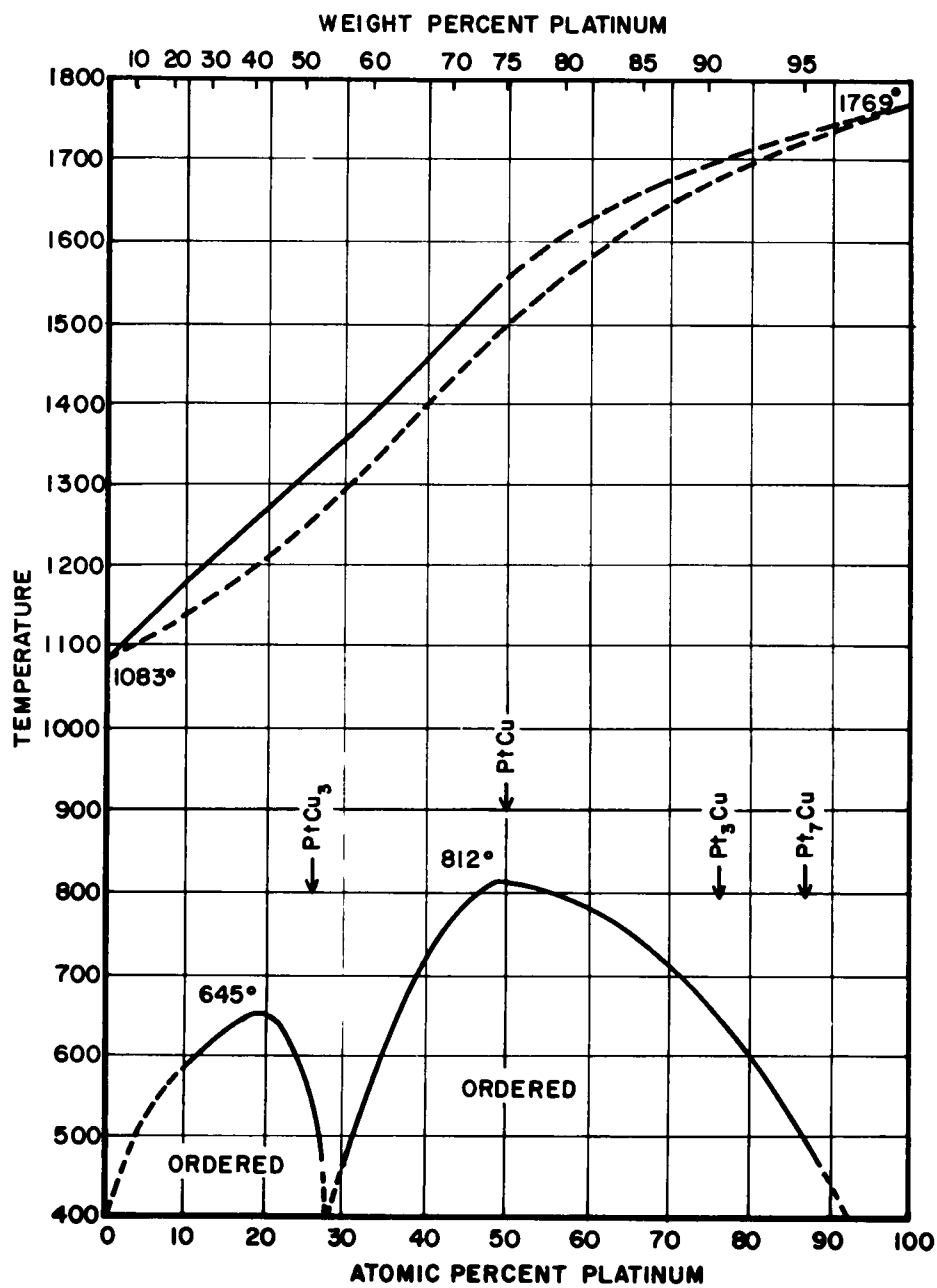


Figure 1. Cu-Pt Phase Diagram

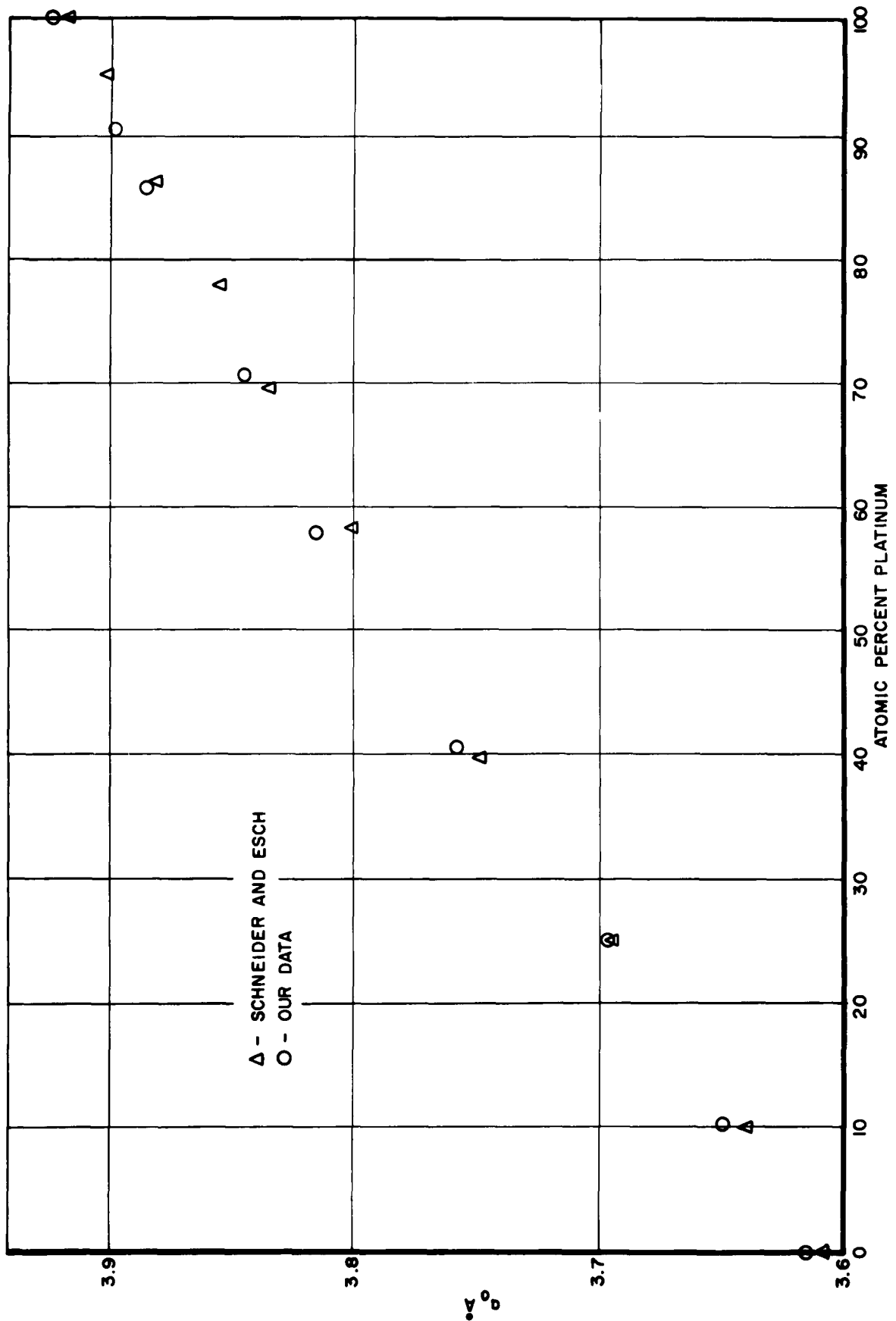


Figure 2. Lattice Parameters ($a_0 \text{ \AA}$) for Cu-Pt Alloys

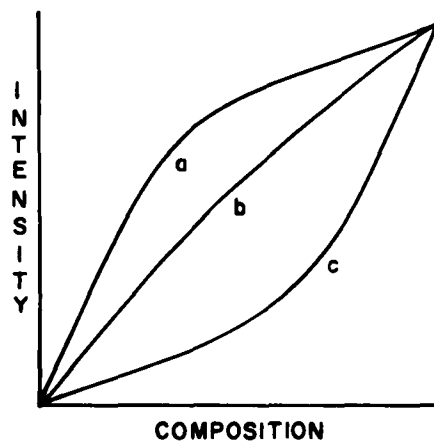


Figure 3. Intensity Versus Composition (General)

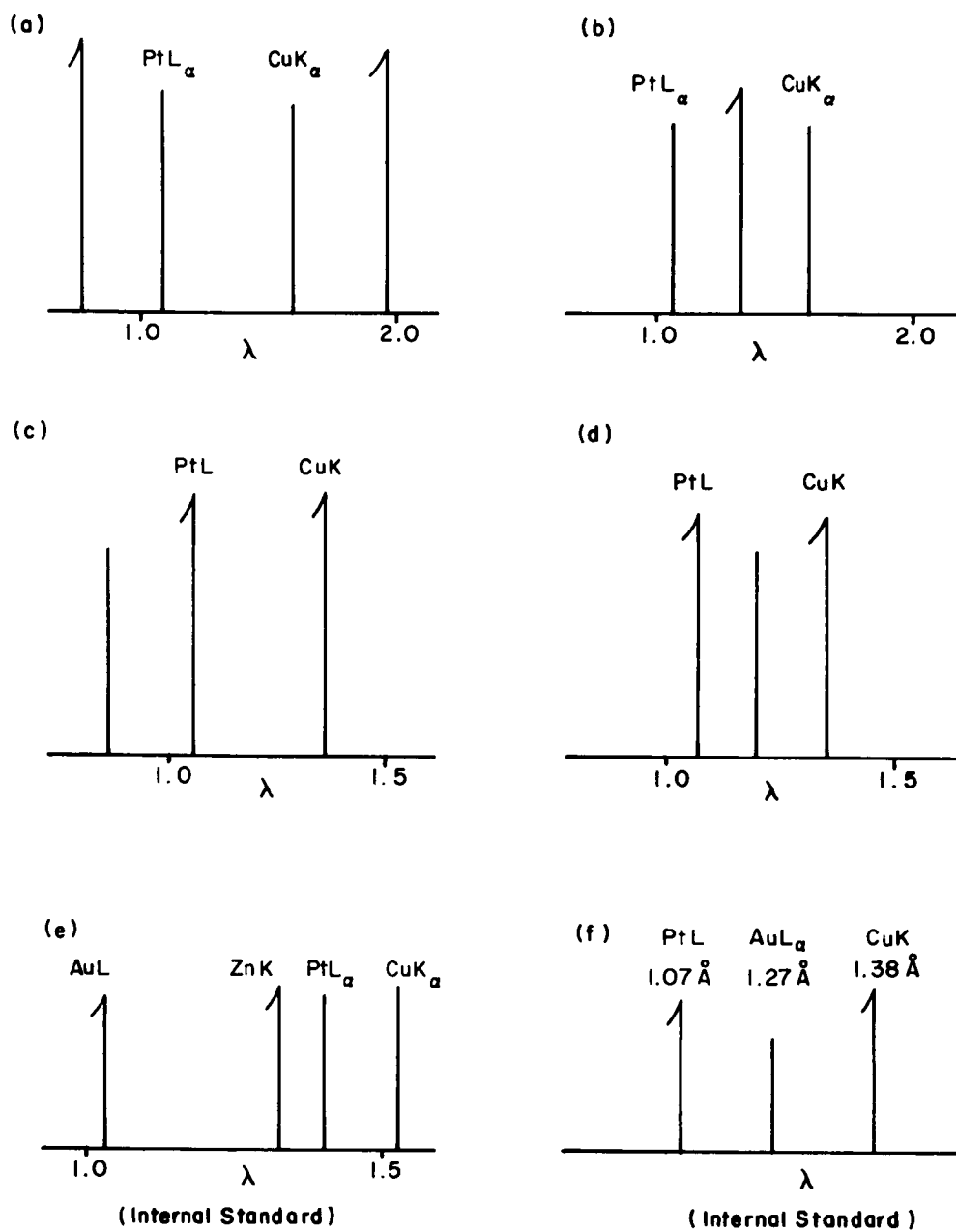


Figure 4. Absorption Edge Considerations for Cu and Pt

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